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Methyl 11,2,3,3a-5a-hydroxy-1-methyl-10-oxo-4-phenyl 10*H*-indeno[1,2-*b*]furo-[3,4-*b*]pyrrole-3a-carboxylate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.043
wR factor = 0.131
Data-to-parameter ratio = 17.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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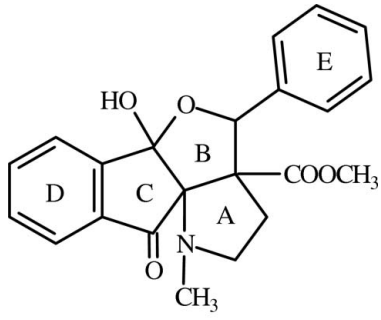
In the title compound, $C_{22}H_{21}NO_5$, the pyrrolidine ring and the five-membered ring in the indene group have envelope conformations, while the furan ring adopts a twist conformation. Weak intermolecular C—H···O interactions link the molecules into centrosymmetric dimers. The crystal packing is further stabilized by van der Waals forces.

Comment

Highly substituted pyrrolidines have attracted much interest in the past few years, since they constitute the main structural element of many alkaloids and pharmacologically active compounds (Subramaniyan & Raghunathan, 2001). Pyrrolidine derivatives inhibit α-mannosidase activity and growth of human glioblastoma and melanoma cells (Fiaux *et al.*, 2005). These derivatives also exhibit anti-influenza virus activity (Stylianakis *et al.*, 2003). Furan derivatives can promote immune activity, or inhibit immune activity and blood platelet aggregation (Li *et al.*, 2005). In view of its medicinal importance, the crystal structure determination of the title compound, (I), was carried out by X-ray diffraction.



(I)

A displacement ellipsoid plot of (I) is shown in Fig. 1. It contains four ring systems, *viz*. pyrrolidine (*A*), furan (*B*), indene (rings *C* and *D*) and phenyl (*E*). The bond lengths in the pyrrolidine ring are comparable with those observed in related structures (Abdul Ajees *et al.*, 2002; Selvanayagam *et al.*, 2004). The sum of the angles around atom N1 (344.3°) is in accordance with sp^3-hybridization.

The methoxycarbonyl group (C15/O4/O5/C16) is planar, with a maximum deviation of 0.006 (1) Å for atom O5. The C—O bond of the ester group is in a *syn* orientation. The torsion angle C16—O5—C15—O4 is 2.8 (2)°. The methoxycarbonyl group and phenyl ring *E* make a dihedral angle of 76.8 (1)°.

Ring *A* adopts an envelope conformation, with puckering parameters q_2 = 0.362 (2) Å and φ = −59.6 (2)° (Cremer & Pople, 1975). Atom C3 deviates by 0.521 (1) Å from the least-

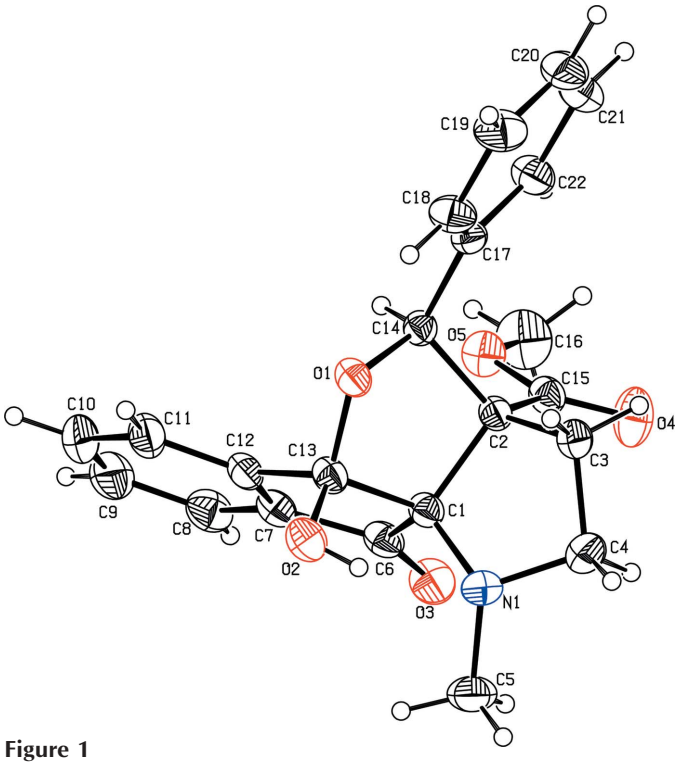


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

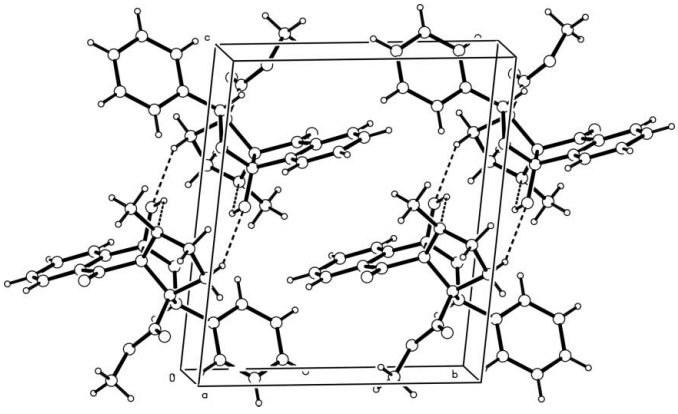


Figure 2
The molecular packing of (I), viewed approximately down the *a* axis. Dashed lines denote O—H···N and C—H···O hydrogen bonds.

squares plane N1/C1/C2/C4. Ring *B* adopts a twist conformation, with puckering parameters q_2 = 0.517 (2) Å and φ = −121.2 (2)°, and displacement asymmetry parameters Δ_S(C13) = 0.086 (1) and Δ_2(C14) = 0.032 (1) (Nardelli, 1983).

The five-membered ring of the indene group adopts an envelope conformation, with puckering parameters q_2 = 0.196 (2) Å and φ = 170.0 (3)°. Atom C6 deviates by 0.118 (1) Å from the least-squares plane C1/C7/C12/C14.

An intramolecular O—H···N hydrogen bond is observed in (I) (Table 2). Weak intermolecular C—H···O interactions (Table 2) link the molecules into centrosymmetric dimers. The crystal packing (Fig. 2) is further stabilized by van der Waals forces.

Experimental

To a refluxing solution of ninhydrin (1 mmol) and sarcosine (1 mmol) in methanol was added methyl 3-hydroxy-α-methylene-3-phenylpropanate (1 mmol). The completion of the reaction was monitored by thin-layer chromatography and the solvent was evaporated under reduced pressure. The crude products were purified by column chromatography and eluted with a hexane–ethyl acetate (9:1) mixture to afford the title compound. The compound was recrystallized from a hexane–ethyl acetate (1:1) mixture as diffraction quality crystals.

Crystal data

$C_{22}H_{21}NO_5$
M_r = 379.40
Triclinic, $P\overline{1}$
a = 8.6216 (5) Å
b = 10.3055 (6) Å
c = 11.7758 (6) Å
α = 79.462 (1)°
β = 79.377 (1)°
γ = 68.876 (1)°
V = 951.48 (9) Å^3
Z = 2
D_x = 1.324 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5288 reflections
θ = 2.5–26.5°
μ = 0.09 mm^{-1}
T = 293 (2) K
Block, colourless
0.24 × 0.22 × 0.19 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
10895 measured reflections
4331 independent reflections
3820 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 28.0°
$h = -11 \rightarrow 11$
$k = -13 \rightarrow 13$
$l = -15 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.131
S = 1.04
4331 reflections
255 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.077P)^2 + 0.1473P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.457 (2)	C1—C2	1.561 (2)
N1—C1	1.458 (2)	C2—C3	1.534 (2)
N1—C4	1.459 (2)	C3—C4	1.509 (2)
C5—N1—C1	119.0 (1)	C1—N1—C4	109.7 (1)
C5—N1—C4	115.6 (1)		
C16—O5—C15—O4	2.8 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3B···O2^{i}	0.97	2.45	3.317 (2)	148
O2—H2···N1	0.82	2.15	2.636 (2)	118

Symmetry code: (i) $-x, -y+2, -z+1$.

The H atoms were positioned geometrically and treated as riding on their parent C atoms, with C—H = 0.93–0.98 Å, O—H = 0.82 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H and 1.2U_{eq}(C or O) for the other H atoms.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine

structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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